Characterization of stratified media using high-resolution thin film measurement techniques

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Abstract. The characterization of stratified media has become essential in the development of industrial applications such as LEDs, solar cells, medical devices, MEMs, MOEMS, etc. This characterization involves measuring the thickness and optical constants of thin layers with high lateral and vertical resolutions. For this purpose, we integrated a spectroscopic reflectometer into an existing 3D optical profiler. We also analyzed how the numerical aperture affects the thickness measurements.

Keywords: stratified media, thin film, 3D optical profiler, spectroscopic reflectometry, numerical aperture.

1. Introduction

Stratified media are obtained from the superposition of micro or sub-micrometric layers of dissimilar transparent materials onto a substrate. Currently, these media are used in many industries, such us semiconductors, flat panel displays, data storage, optical coatings and medical devices.

Research activities and industrial quality control applications require the accurate characterization of a range of properties of stratified media, including film thickness, refractive index and 3D surface shape of the upper and lower interfaces of the film.

Optical techniques are suitable for measuring and characterizing stratified media. Due to the refractive index variation in the axial direction, UV, visible and IR light is reflected in the various interfaces. As a result, reflected wavefronts are superposed to give rise to interference patterns and/or changes in polarization.

The following four techniques are available in order to measure film thickness. Optical profiling techniques, mainly confocal and vertical scan interferometry (VSI), are suitable for characterizing transparent thick films [1]. Measurements are carried out by vertically scanning the sample through the upper and lower film interfaces. The thickness of the layer is determined by the two peaks in the confocal axial response or by the two sets of interference fringes during the vertical scan, respectively. The 3D topographies of the upper and lower interfaces of the film can also be obtained. Confocal can be used when the axial response shows two well-resolved peaks at the two parallel reflecting interfaces. The width of the peaks is reduced when the NA of the objective increases and the distance between the peaks increases with the thickness of the film. In interferometry, the width of the correlograms is reduced by using a broad band light source with a short coherence length. From a practical point of view, the thinnest film that can be measured is 1 μm for a white light interferometer and close to 1.5 μm when a confocal profiler is used. Both techniques have very good lateral resolution, better than 0.5 μm.

The third technique is spectroscopic reflectometry, in which the reflected light intensities are measured in a broad wavelength range. The thinnest film that can be measured is 10 nm. The weakness of this technique is that the lateral resolution depends on the diameter of the optical fiber that is being used, which is usually greater than 100 µm.
The last technique is ellipsometry, which is based on an analysis of the change in polarization of the light that is reflected on the surface of the sample. In this technique, the vertical resolution that can be achieved is 0.1 nm and many layers can be measured in stratified media. However, the lateral resolution is even worse than that of spectroscopic reflectometry.
Several applications such as microsensors, micromembranes, LEDs, solar cells, implants, hemodynamic devices, MEMs, MOEMs, etc. require high lateral and vertical resolution. An ideal system for characterizing stratified media should offer the lateral resolution of an optical profiler as well as the vertical resolution of a spectroscopic reflectometer.
The goal of this master's degree project is to integrate a spectroscopic reflectometer into a 3D optical profiler, and analyze the limitations that are related to the numerical aperture (NA) of the microscope objectives. We will also use this new configuration to characterize some real examples of stratified media.
To our knowledge, this is the first time that a spectroscopic reflectometer has been integrated into a 3D optical profiler.

2. State of the art

A film of a certain thickness and optical constants (n,k) may behave as thick or thin in an optical profiler, depending on the NA of the objectives and the coherence length of the light source. A film is considered to be thin when the two peaks of the confocal axial response or the two VSI correlograms become unresolved.
In confocal mode, the limit depends only on the NA: the higher the NA, the narrower the axial response. Therefore, the same film may behave as thick for low NA (peak resolved) and thin for higher NA (peak unresolved). In VSI techniques, the width of the correlograms depends on the coherence length of the light source.
Spectroscopic reflectometry is a technique for fast and accurate measurement of a thin film structure. It works very well for single thin layers on a substrate, single foils or membranes, and can also deal with more sophisticated structures (usually up to ten layers on a substrate).
In spectroscopic reflectometry, the surface is illuminated with a perpendicularly incident broadband light beam. Light reflected at the different interfaces is superposed coherently and interferential effects occur. As a result, the intensity of the total reflected light shows variations in wavelength that depend on the thickness and optical constants of the different layers of the stratified media.
Reflected light is propagated via an optical fiber to a spectrometer. In the spectrometer, the spectrum of the reflected light is recorded. Software compares the real measured spectrum with a simulated one and optimizes the thickness of the thin films until the best fit is achieved. This is called an indirect method, as the result (the thickness and the optical constants) is derived from a comparison between a measurement and a model.
Ellipsometry is a much more sophisticated technique than spectroscopic reflectometry. It is also an indirect method and is based on an analysis of the polarization of the light reflected by a thin film structure.
The main advantages over spectroscopic reflectometry are that multilayer structures (up to 30 layers on a substrate) can be assessed and extremely thin films can be measured. The lower limit for spectroscopic reflectometry is 10 nm, while it is 1 nm for ellipsometry.
There are a few drawbacks to ellipsometry in comparison with spectroscopic reflectometry. The optical setup cannot be perpendicular to the surface, it has to be illuminated and observed with a very high angle of incidence. Alignment is very difficult, and cannot be integrated into an optical profiler or a control of production system. Basically, ellipsometry is a sophisticated technique that is used in research labs.

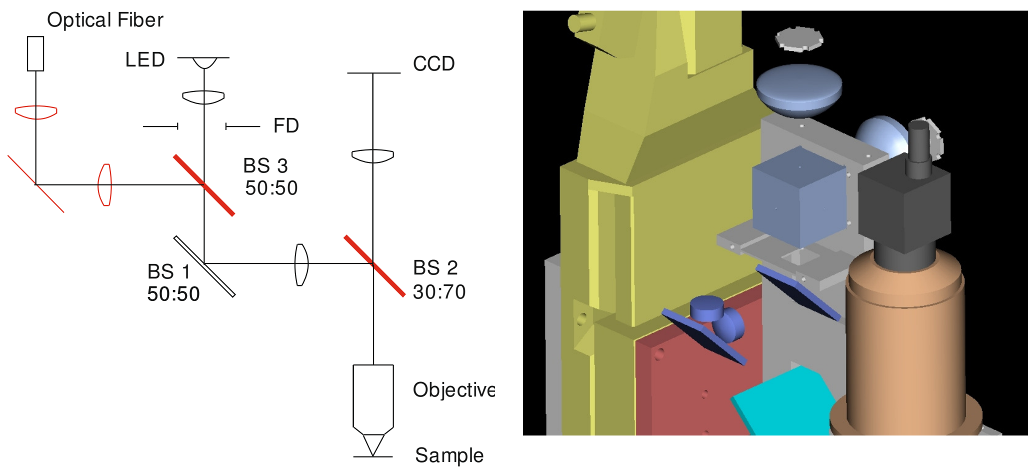
Table 1. Lower Limit and vertical and lateral resolutions.

Technique	Lower Limit (μm)	Vertical resolution (nm)	Lateral resolution (μm)
Confocal	1.5	1	0.3
Interferometry (VSI)	1	1	0.5
Spectroscopic reflectometry	0.01	0.1	>100
Ellipsometry	0.001	0.1	>100

The main difficulty of integrating a spectroscopic reflectometer into a 3D optical profiler is characterizing the effect of changing the optical path that is induced by the NA of the objectives. This effect has already been analyzed for confocal and VSI techniques. For low NAs, the simple geometrical model of depth distortion can be assumed to be valid. Thus, effects such as apodization, depolarization and aberration can be ignored [2]. However, these effects appear when the NA of the objective is larger than 0.45. Hence, a rigorous model based on the Debye theory needs to be used. Such a rigorous model was developed in a previous study by C. Cadevall [3], in which light bandwidth, dispersive materials, non-uniform illumination and system aberrations were taken into account. This model is reliable for high NAs.
However, to our knowledge, a mathematical model that accounts for the effects of high NAs in spectroscopic reflectometry and ellipsometry has not been developed yet.

3. Conceptual Design

Our aim is to integrate a spectroscopic reflectometer into an existing optical profiler. We will use the PLμ 4300 Optical Profiler from Sensofar-Tech S.L. and the Nanocalc-2000 spectroscopic reflectometer from Mikropak GmbH. Figure 1 (a) shows in red the modifications of the design for integrating the optical fiber. Figure 1 (b) shows a 3D simulation of the final configuration.



(a) (b)

Figure 1. Optical design: (a) final setup, (b) 3D simulation.

The final design differs in three main ways from the original one. Firstly, the second beam splitter 50:50 has been changed to one of 30:70. Figure 2 shows the mathematical calculations for the light distribution. Thus, the light coming back to the illumination path is almost double. And the light striking the CCD camera is only reduced from 25% to 21%.

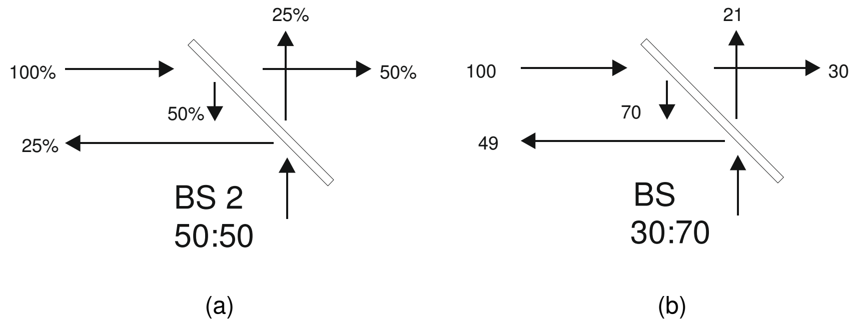


Figure 2. Beam splitter light distribution: (a) 50:50, (b) 30:70.

The second modification was to add a third beam splitter 50:50 to the illumination path. Finally, a relay was added and a 45-degree mirror, which allows the field diaphragm to move to a vertical position. The optical fiber could not be placed horizontally for mechanical reasons related to the original cover design.

Figure 3 (a) and (b) shows the new optical design simulated with ZMAX with two different objectives. One remarkable aspect of this design is that the spot size on the sample changes with the objective but the spot size on the CCD camera remains the same, at 32 µm.

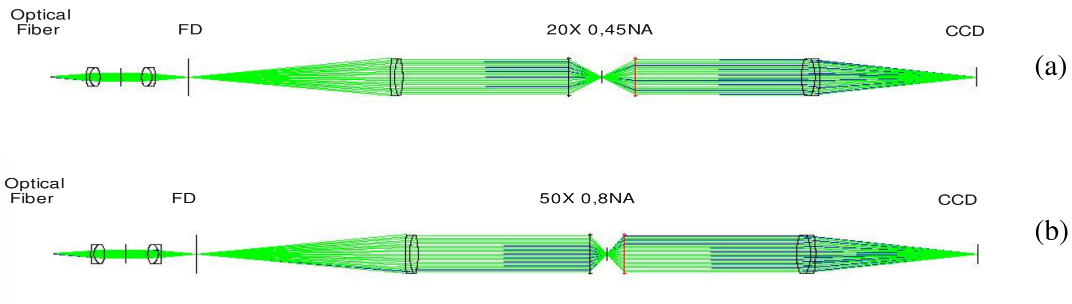


Figure 3. Optical design simulation using (a) a 20X 0.45NA objective; (b) a 50X 0.8NA objective.

The calculation of the spot size on the sample is given by:

$$h = \frac{200 * \emptyset}{125 * M}$$

Where:

Ø = Diameter of the optical fiber

M= Objective magnification

Table 2 shows the spot size of some objectives using a 100 µm optical fiber.

Table 2. Spot size calculations.

Magnification	NA	Spot Size (µm)
5XEPI	0.15	32
10XEPI	0.30	16
20XEPI	0.40	8
50XEPI	0.8	3.2

4. Prototype development

Figure 4 shows a detailed 3D opto-mechanical model of the test bench. Some of the main innovations of the multifunction optical Sensorhead are: a double light source (with white and monochromatic LEDs), a closed loop vertical scanning stage with a large travel range and a positioning repeatability of 0,2 µm, and a PZT of 200 µm of travel range with a repeatability of 1 nm and a linearity of 0.05%. The system is flexible, as it can work with different field lenses (from 0.5X to 2X) and with CCD cameras with different formats, resolutions, and speeds. Finally the design is compatible with all infinite-corrected microscope objectives (Nikon, Leica, Olympus, etc.).

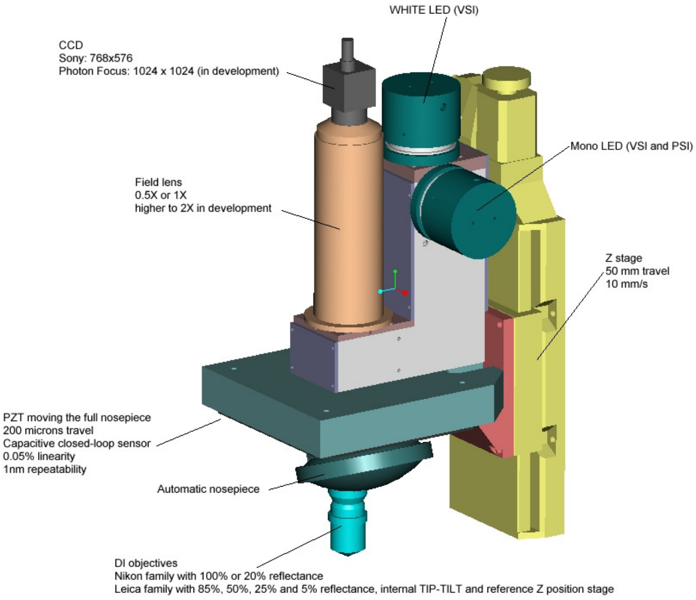
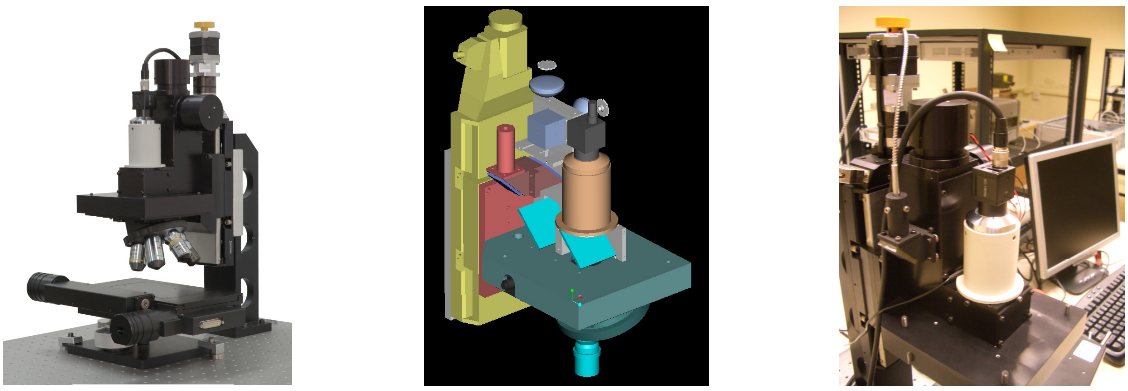


Figure 4. 3D Model of the optomechanical design of the test bench.

Figure 5 (a) shows the experimental setup of the test bench. The multifunction optical Sensorhead is assembled in a vertical stand over an isolation breadboard. Also shown is the nosepiece where the microscope objectives are placed, and an XY stage for positioning the sample. Under the XY stage is a mechanical device for adjusting the sample tip-tilt so that it is perpendicular to the optical axis. This kind of high precision positioning is essential for the PSI interferometric mode, in which vertical repeatability values of 0.01 nm are obtained.

Figure 5 (b) shows the 3D model of the optomechanical design that allows the integration in the test bench of the fiber optic, which is connected to a spectroscopic reflectometer. The experimental assembly of this integration is also shown. Spectroscopic reflectometry enables the multifunction Sensorhead to measure thin transparent and semi-transparent films of 10 nm thickness and to carry out multilayer measurements of up to 4 layers on almost any kind of substrate. In addition, the integration of the spectroscopic reflectometer allows these stratified media to be measured with high lateral resolution, even lower than 10 μm.



(a) (b) (c)

Figure 5. Multifunction Sensorhead: (a) experimental setup, (b) 3D model, (c) integration of the fiber optics connected to the spectroscopic reflectometer.

5. Experiment and results. Influence of the NA of the objectives

To analyze the effect of the NA of the objectives on the spectroscopic reflectometer, we compared the results obtained with different magnifications and NA objectives on a step wafer reference.

The list of objectives used is shown in Table 3.

Table 3. List of objectives used in the experimental tests.

Magnification	NA	Manufacturer	Field of View (mm^2)	Working distance (mm)
5XEPI	0.15	Leica	2.54x1.91	12
10XEPI	0.30	Leica	1.27x0.95	11
20XEPI	0.40	Leica	0.635x0.476	1.15
20XEPI	0.45	Nikon	0.635x0.476	4.5
20XEPI	0.50	Leica	0.635x0.476	1.27
50XEPI	0.8	Leica	0.254x0.190	0.50
50XEPI	0.9	Leica	0.254x0.190	0.28

The reference step wafer is a calibrated Si-SiO of 5 steps (0-500 nm) of 4' diameter. It is a wafer with six distinct areas: one without coating that serves as a reference; and five areas with coatings from 100 to 500 nm. The actual values of each step have been measured with a calibrated ellipsometer. These values and those taken by the spectral reflectometer used in the integration without objectives are shown in Table 4.

Table 4. Nominal and actual values measured with an ellipsometer, and an image of this.

Nominal values (nm)	Ellical (nm)
0	1.25
100	106.55
200	201.45
300	297.24
400	397.27
500	497.26

The settings of the spectroscopic reflectometer Nanocalc 2000 are shown in Table 5.

Table 5. Measurement settings for the spectroscopic reflectometer.

Settings	Values
Analysis wavelengths	425-700 nm
Integration time	400 ms
Boxcar width (pixel)	9
Samples to average	1
Maximum intensity in spectrum	530 nm

The step wafer has 5 different areas of different thicknesses. Figure 6 shows the results obtained for the thickest area, the step area of 497.26 nm.

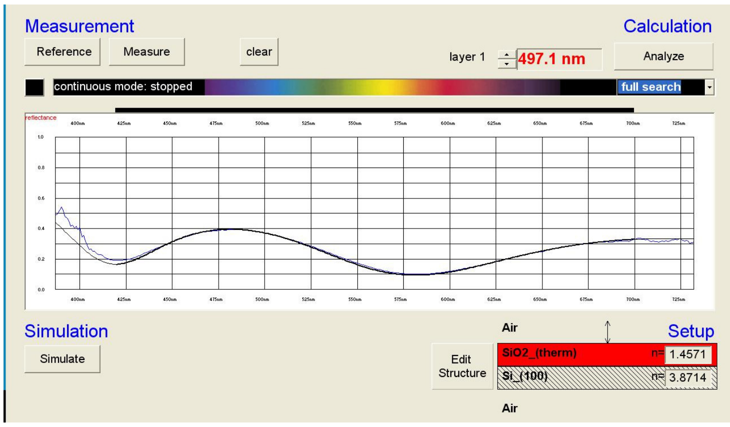
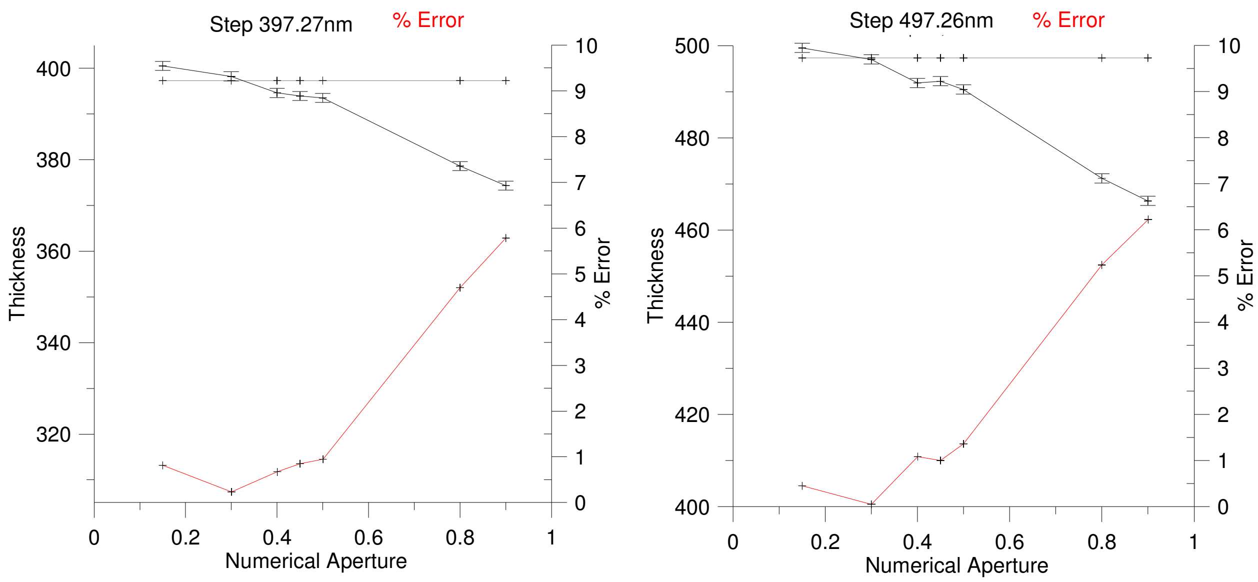


Figure 6. Measurement result with the spectroscopic reflectometer using a 10X 0.3NA objective on a step area of 497.26nm.

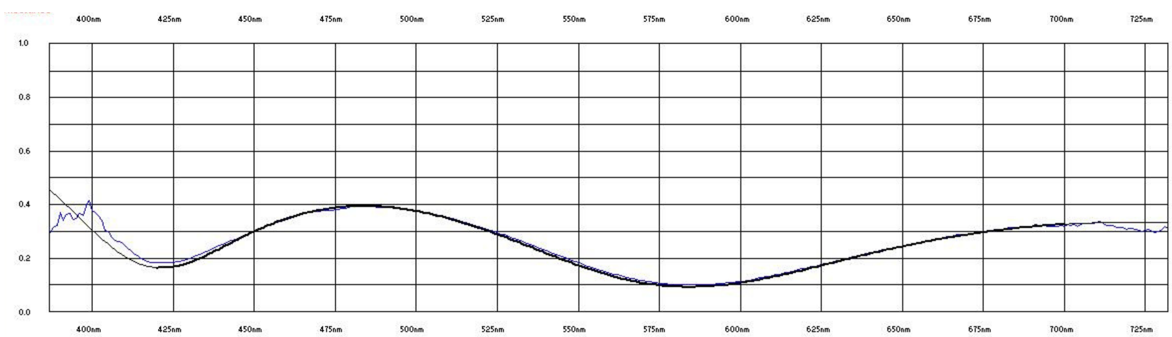
The graphs in Figure 7 show the values obtained for the 397.27 and 497.26 nm step areas and the percentage of error with different NAs.



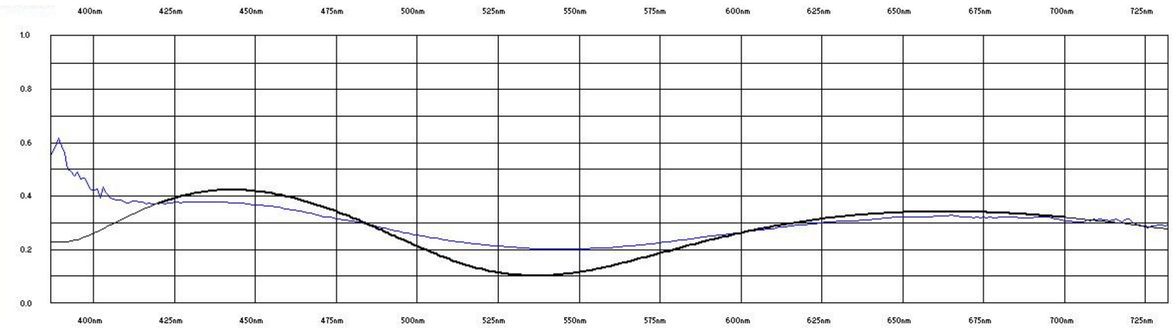
(a) (b)

Figure 7. Graphs showing the measured values and the percentage of error for the different NAs in the 397.27 and 497.26 nm step.

Figure 8 shows different spectral measurements made in the step area of 497.26 nm with very different NA objectives.



(a)



(b)

Figure 8. Graphs showing different spectra in the same sample of 497.26 nm steps size with different NA objectives: (a) 5X 0.15NA and (b) 100X 0.9NA. The X axis represents the wavelength and the Y axis the reflectivity.

Finally, Figure 9 shows changes in the fitness parameter regarding the NA. The manufacturer indicates that a good fitness value is below 0.3.

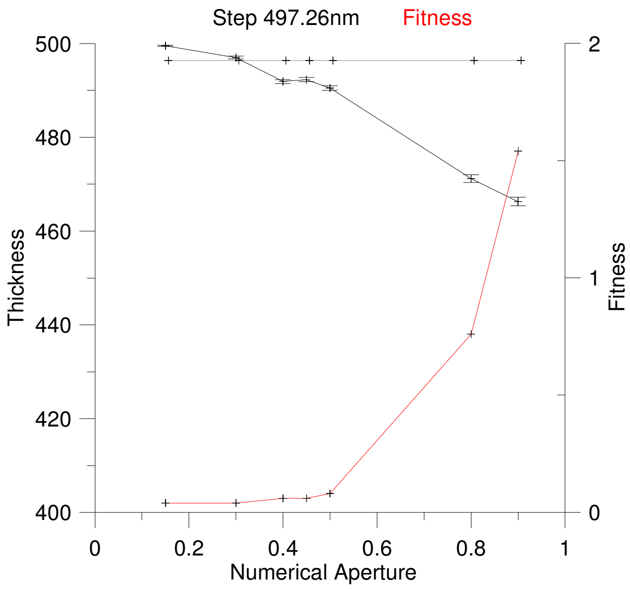


Figure 9. Graph showing the measured values and the fitness parameter for the different NAs for the 497.26 nm step area.

6. Discussion

From the previous experimental results we observed that when the NA of the objectives goes up, the accuracy error of the measurements always increases and the thickness values always decrease. This effect can be observed by analyzing Figure 8, which shows the results for the same sample with two different numerical apertures. Figure 8 (b) shows that the maximum and minimum values of reflectivity have been shifted to the left, that is, towards smaller values of wavelengths. This implies lower values of thickness. When the NA is increased, the condition of constructive or destructive interference is obtained for shorter wavelengths. This is consistent with the rigorous theoretical model developed from the Debye model in C. Cadevall’s doctoral thesis [3].

The evolution of the spectroscopic results for the different steps and the different NAs shows that the higher the NA is, the lower the contrast of the modulation signal of the spectroscopic reflectometer. This effect is also shown in Figure 8. This is because the light beam is not perpendicular to the sample as in a standard configuration of a spectroscopic reflectometer. Now we have light beams with different angles of incidence and different optical paths. This effect increases the range of possible results and therefore decreases the reflectivity contrast. The fitness parameter shows this effect in Figure 9.

7. Real applications

7.1. Microphone diaphragm thickness

Samples are silicon microphone devices for pressure sensor application. Figure 10 (a) shows the topography in VSI of the full device. Figure 10 (b) shows the topography in PSI mode of the membrane. The goal is to measure the thickness distribution of the Si diaphragm.

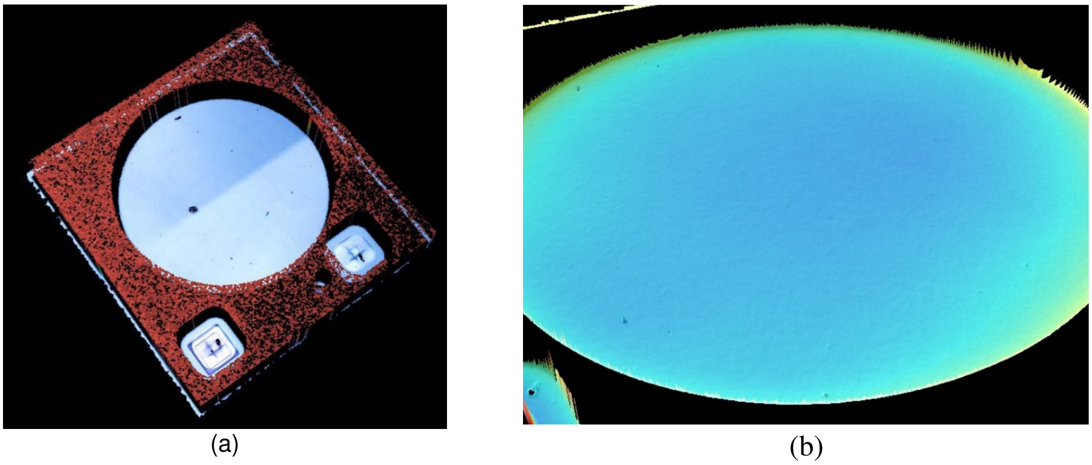


Figure 10. (a) Plan view, (b) Cross-section.

The application needs a high-resolution thin film measurement, as the thickness values are around 1 µm and the customer needs to know the thickness distribution throughout the whole diaphragm, which implies high lateral resolution. In this case, an optical fiber of 200 µm and a 10X objective have been used to obtain a spot size of 40 µm. Figure 11 shows two different positions of the sample for the measurements and Table 6 shows the results.

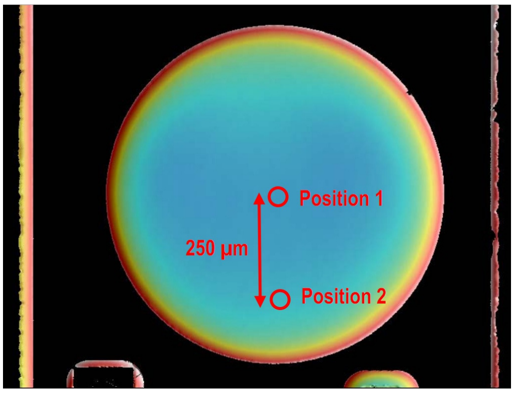


Figure 11. Two positions of the membrane.

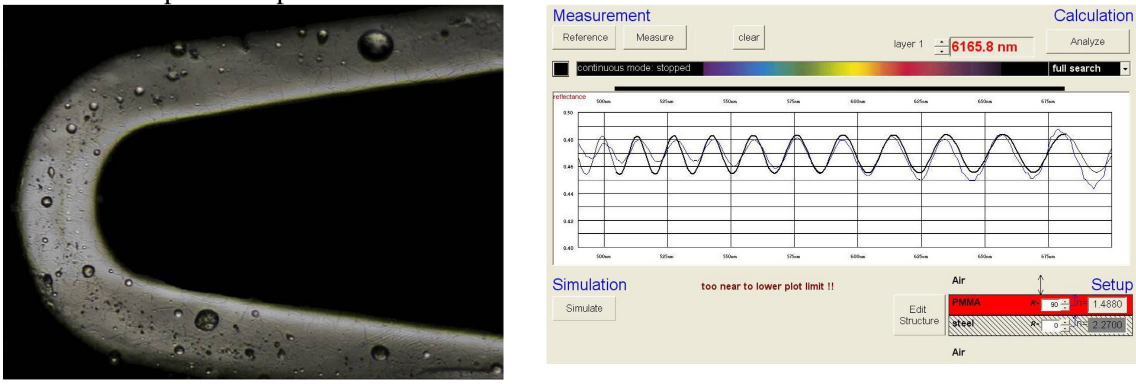
Table 6. Thickness measurements in positions 1 and 2.

Position	Thickness (nm)
1 Center	968.7
2 Bottom	972.4

7.2. Stents

The measurement of stents is one of the examples that best illustrates the main advantages of the new high-resolution thin film measurement techniques. Stents are made of steel, but also carry small amounts of compounds that are toxic to the human body. One of the options for solving this problem is to coat the steel with biocompatible material. Manufacturers need to characterize the thickness of the coating to prevent irreversible damage. However, this is very difficult, as the surface is rough and curved and the thickness ranges from 0.5 to 7 µm. The new setup that focuses the light beam of the spectroscopic reflectometer and reduces the spot size in the sample causes rough and curved samples to behave as if they were smooth and flat.

The sample used in this case is a stent coated with a polyurethane acrylate layer [5]. In some cases, drugs are also added, but this is not the case here. Specifically, this material was not in the reflectometer database, after consultation with the manufacturer, we concluded that the optical properties were similar to those of PMMA. Figure 12 shows an image of the analysis area and the spectroscopic result. The thickness in this area is 6165.8 nm.



(a) (b)

Figure 12. (a) Image of a stent area acquired with a 20X, (b) spectroscopic result.

8. Conclusions

The integration of a spectroscopic reflectometer into an existing 3D optical profiler has been carried out successfully. This system can characterize stratified media with high lateral and vertical resolution. The new configuration can solve thin film applications, which was not possible previously. In this master's degree project, two applications of considerable importance have been resolved successfully: thickness measurements of coatings in stents and microphone membranes.

Thus, the new system configuration will lead to advances in the optical metrology surface field, at micrometric and nanometric scale. A new, high performance optical Sensorhead has been designed.

The limitation of this new configuration is given by the NA of the objectives. For NAs that are less than 0.3, the accuracy error of the measurements is negligible and the results are considered correct. However, for values between 0.3 and 0.5, the accuracy errors are between 1 and 2%. Finally for NAs above 0.5, the measure is no longer valid and the errors that are obtained increase dramatically.

9. Acknowledgments

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References

[1] C. Cadevall, C. Oriach-Font, R. Artigas, A. Pintó and F. Laguarta, "Improving the measurement of thick and thin films with optical profiling techniques", SPIE, Vol. 6166, 66161Z, 2007.
[2] M. Gu, Advanced Optical Imaging Theory, Springer Verlag, 2000.
[3] C. Cadevall, New developments in optical surface profiling, PhD Thesis, UPC, 2007.
[4] C. Oriach-Font, F. Laguarta, C. Cadevall, R. Artigas, and A. Pintó, "Deep 3D Optical Metrology", Focus on Microscopy, Valencia.
[5] A. Srivastava, D. Agarwal, S. Mistry, J. Singh, "UV curable polyurethane acrylate coatings for metal surfaces", Pigment & Resin Technology, Vol. 37, Issue 4, Page 217-223, 2008.
[6] C. Cadevall, R. Artigas, and F. Laguarta, "Development of confocal-based techniques for shape measurements on structured surfaces containing dissimilar materials", SPIE, Vol. 5144, pp 206-217, 2003.
[7 C. Oriach-Font, F. Laguarta, C. Cadevall, R. Artigas, and A. Pintó, "Deep 3D Optical Metrology", Focus on Microscopy, Valencia, 2007.
[8] Eszter Bognár, György Ring, Hilda Zsanett Marton, János Dobránszky and János Ginsztler. "Polyurethane Coating on Coronary Stents", Key Engineering Materials Vols. 345-346 (2007) pp. 1269-1272.